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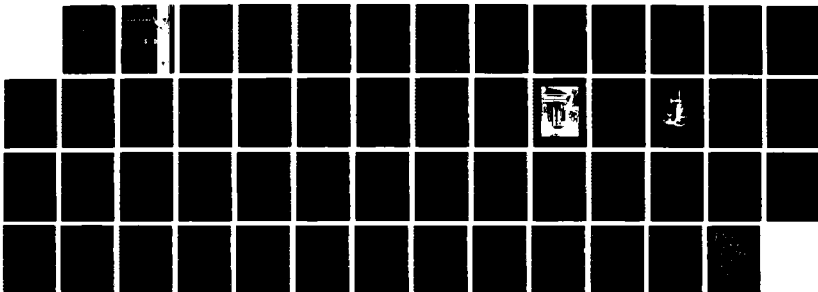
LABORATORY TESTING OF A FLUIDIZED BED DRY SCRUBBING
PROCESS FOR REMOVAL OF (U) OAK RIDGE INST OF NUCLEAR
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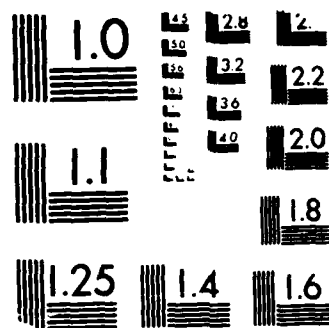
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Final Report

USATHAMA

U.S. Army Toxic and Hazardous Materials Agency

LABORATORY TESTING OF A
FLUIDIZED BED DRY SCRUBBING PROCESS
FOR REMOVAL OF SULFUR DIOXIDE AND
PHOSPHOROUS PENTOXIDE FROM AN
INERT CARRIER GAS

MARCH 1988

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Prepared by

Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831
operated by
MARTIN MARIETTA ENERGY SYSTEMS, INC.
for the
U.S. DEPARTMENT OF ENERGY
Under Contract No. DE-AC05-84OR21400

Prepared for

U.S. Army Toxic and Hazardous Materials Agency
Aberdeen Proving Ground, Maryland

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EXECUTIVE SUMMARY

The use of a fluidized bed contactor to scrub acidic gases generated in waste material incineration appears to be a viable means of treating flue gas. The basic difference between dry scrubbing and conventional wet scrubbing techniques is that dry scrubbing does not result in an aqueous waste. This difference should prove to be very advantageous at an incineration facility that does not already have an adequate wastewater treatment facility or a facility that does not have an abundant supply of process water.

PROGRAM OBJECTIVES

Oak Ridge National Laboratory (ORNL) conducted a series of bench-scale tests to determine the feasibility of using dry scrubbing to remove acidic gases commonly produced by hazardous waste incinerators. The primary waste gases considered in this study are phosphorous pentoxide (P_2O_5), sulfur dioxide (SO_2), and hydrogen chloride (HCl). Acidic gases, such as nitrogen oxides (NO_x) and hydrogen fluoride (HF), may also be present in incinerator flue gas but were not included in this study.

The experimental program was divided into two parts: the first, covered in this report, was designed to answer basic questions concerning the feasibility of removing acidic gases by dry scrubbing; the second will focus on issues relating to the economics of the process, as well as on a full bench-scale demonstration of the process.

The specific objectives of the experimental program covered in this report were to

1. determine the feasibility of removing P_2O_5 in a fluidized bed of alkaline material,
2. determine an "operating envelope" for the dry scrubbing of P_2O_5 , and
3. verify that SO_2 can be scrubbed at the same process conditions.

The specific objectives of the second phase of the experimental program are to

1. verify that HCl can be scrubbed at the same process conditions,
2. screen candidate sorbents,
3. determine scrubber performance with a mixture of acidic gases that represents a typical flue gas composed of multiple contaminants, and
4. obtain data necessary to evaluate the economics of the dry scrubbing process and establish design criteria for a pilot-scale scrubber.

SUMMARY

It is feasible to remove P₂O₅ and SO₂ in a fluidized bed consisting of calcium oxide operating in a bubbling bed regime at 540°C (1000°F). No adverse "coeffects" between P₂O₅ and SO₂ exist when they are scrubbed simultaneously.

Of the parameters considered, gas velocity had the most significant effect on efficiency of P₂O₅ removal, probably because vigorous bubbling in the bed resulted in better solid-gas contacting. The removal efficiency is greater at 3.5 times the minimum fluidization velocity than at 1.8 times the minimum fluidization velocity. Removal efficiency for P₂O₅ increases with temperature, probably because of an increase in the reaction rate constant. The removal efficiency is slightly greater at 540°C (1000°F) than at 480°C (900°F) (i.e., 99.8 vs 99.7%).

CONCLUSIONS AND RECOMMENDATIONS

Dry scrubbing is a viable method for treating acidic gases produced by incineration. Site-specific data — such as the capacity and capability of the wastewater treatment plant, availability of sorbent material in the local area, and availability of process water — must be considered to directly compare dry scrubbing with other air pollution control technologies.

The removal efficiencies measured for P₂O₅ and SO₂ exceeded 99% at all conditions studied. The bench-scale dry scrubbing program should be continued to obtain the data necessary to compare the economics of dry scrubbing with other technologies.

LABORATORY TESTING OF A FLUIDIZED BED
DRY SCRUBBING PROCESS FOR REMOVAL OF
SULFUR DIOXIDE AND PHOSPHOROUS PENTOXIDE
FROM AN INERT CARRIER GAS

W. M. Bradshaw

1. PROGRAM OVERVIEW

The U.S. Army, which is currently working on remedial environmental programs at several installations, has found a wide variety of wastes in soil and in settling ponds. The contaminated sludges, solids, and liquids must be disposed of definitively.

One of the most preferred disposal alternatives for hazardous materials is incineration, and the military routinely incinerates waste sludges, soils, and liquids. However, these wastes usually contain chemical compounds (and their derivatives) that will produce acidic gases during incineration.

Incineration (under oxidizing conditions) of compounds containing sulfur, phosphorus, or chlorine will produce sulfur dioxide (SO_2), phosphorus pentoxide (P_2O_5), and hydrogen chloride (HCl), respectively. In addition, nitrogen oxides (NO_x) will be formed from both the incineration of compounds containing nitrogen and the oxidation of atmospheric nitrogen at temperatures encountered in incineration systems. In most cases, these gases must be scrubbed from the flue gas to meet environmental standards.

The U.S. Army Toxic and Hazardous Materials Agency (USATHAMA) is evaluating alternative methods of scrubbing acidic gases from flue gas. This report documents experimental work on a dry scrubbing process that uses a fluidized bed of quicklime (calcium oxide) to remove acidic gases from a nitrogen carrier gas stream at 480 to 650°C (900 to 1200°F).

The bench-scale experimental program consists of a 2-year effort that will provide the data necessary to evaluate the relative costs of dry and wet scrubbing and to design a pilot unit. The first phase of the bench-scale experimental program focused on determining the feasibility of dry scrubbing P_2O_5 and SO_2 in a fluidized bed. The second

phase of the program will consider (1) the dry scrubbing of HCl, (2) bed performance with a mixture of gases that model flue gas, and (3) sorbent utilization.

The objectives of the bench-scale experimental program are to

1. determine the feasibility of removing P₂O₅ in a fluidized bed of alkaline material,
2. determine an "operating envelope" for the dry scrubbing of P₂O₅,
3. verify that SO₂ can be scrubbed at the same process conditions,
4. verify that HCl can be scrubbed at the same process conditions,
5. screen candidate sorbents,
6. determine scrubber performance with a mixture of acidic gases that represents a typical incinerator flue gas, and
7. obtain data necessary to evaluate the economics of the dry scrubbing process and design a pilot-scale scrubber.

This report covers the first three objectives. The remaining objectives will be addressed in the FY 1988 experimental program.

2. BACKGROUND

For this study, "dry scrubbing" is defined as a process that does not produce a liquid waste. The conventional wet scrubbing process typically uses an adiabatic water quench, followed by an inertial scrubber to remove particulates, and a packed bed scrubber to remove acidic gases. Dry scrubber systems are not as well defined, but all dry scrubbing systems use a gas-solid or gas-slurry contacting device and a particulate removal system to remove the solid residue. Dry scrubbing is becoming a viable alternative to the more conventional wet scrubbing process.

2.1 ASSESSMENT OF DRY SCRUBBING TECHNOLOGY

A considerable amount of research has been conducted by the electrical power generation industry on dry flue gas desulfurization. A number of lab-scale studies have been conducted in this area, dating back to the late 1960s.¹ In 1982 the Electric Power Research Institute (EPRI) and the Public Service Company of Colorado (PSCC) successfully demonstrated a dry sorbent injection system on a 22-MW boiler burning low-sulfur coal.²

Dry scrubbing was first applied to hazardous waste incinerators in the form of a spray dryer in 1982 at the Nitro Atomizer plant in Kommuekemia, Denmark.³ Since that time, dry scrubbing has been applied at several regional, noncommercial incinerators in Europe. The typical capacity of these incinerators is in the range of 100 to 130×10^6 Btu/h (29 to 38 MW).³

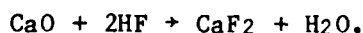
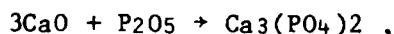
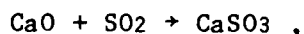
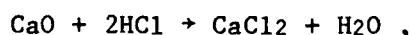
Dry scrubbing is not limited to incineration systems. Oak Ridge National Laboratory has successfully removed hydrogen fluoride (HF) from vent gas streams in a bench-scale packed bed reactor using quicklime at 135 to 300°C.⁴ A 99% HF removal rate was reported at calcium utilizations approaching 60%.

2.2 COMPARISON OF WET AND DRY SCRUBBING

The most significant advantage of dry scrubbing is that no liquid effluents have to be treated. Although wastewater treatment may not be a significant problem for a large chemical plant, it can amount to a substantial fraction of the cost of an incineration facility. Because dry scrubbing eliminates the need for wastewater treatment, it has become a viable option for treatment of flue gas from incinerators.

Another advantage of dry scrubbing is that it can use calcium-based (alkaline) sorbents. Conventional wet scrubbers used on many hazardous waste incinerators typically use alkali (sodium-based) chemicals to neutralize acidic gases. Alkali materials are ~8.5 times more expensive than pebble lime (on a molar equivalent basis).³ If large quantities of acid are to be removed, the alkaline materials can provide a significant cost savings in reagent costs alone.

A corollary benefit of using alkaline sorbents is that many of the waste products (e.g., CaSO_3 , $\text{Ca}_3(\text{PO}_4)_2$, and CaF_2) are insoluble in water. This is of particular benefit if the spent sorbent is to be disposed of in a landfill. The specific reactions between acidic gases and an alkaline sorbent such as lime (calcium oxide) are



Dry scrubbing processes do not result in a gas stream that is saturated with water, thus reducing or eliminating plume opacity and in some cases, reducing corrosion of downstream equipment.

Another advantage of dry scrubbing is that it requires relatively small amounts of process water. A limited quantity of water would still be required to quench the afterburner exhaust to reduce the flue gas temperature, but that is a small fraction of the water required for wet scrubbing. Availability and cost of process water are potential problems for large incinerators located in the western United States.

Finally, the solid effluent resulting from the incineration of Resource Conservation Recovery Act (RCRA)-defined hazardous wastes possibly could be delisted, thus allowing the solid residue to be disposed of in a solid-waste rather than a hazardous-waste landfill. The potential for delisting is dependent on the particular incineration process because adsorption of heavy metals and products of incomplete combustion (PICs) by sorbent particles has been reported.³ If the heavy metals or PICs are captured by the sorbent, delisting would depend on leaching characteristics.

2.3 TYPICAL DRY SCRUBBING PROCESS

A diagram of a typical dry scrubbing system is shown in Fig. 2.1. The system shown uses an entrained bed as a primary contactor, followed by a cyclone for solids removal, dilution air for cooling, and a baghouse to remove particulates. The baghouse also provides additional residence time for acidic gas-sorbent contacting, thus improving the acid removal rate.

Several alternatives are available to the system presented in Fig. 2.1. Most notably, several dry scrubbing processes combine the quench and the contactor into a spray dryer, a common practice in the municipal waste incineration industry. Of secondary importance is the replacement of the dilution air system with a gas cooler. Although an electrostatic precipitator could be used in place of the baghouse, a baghouse is preferred because 15 to 20% of the acid is removed by the sorbent that coats the bags.³

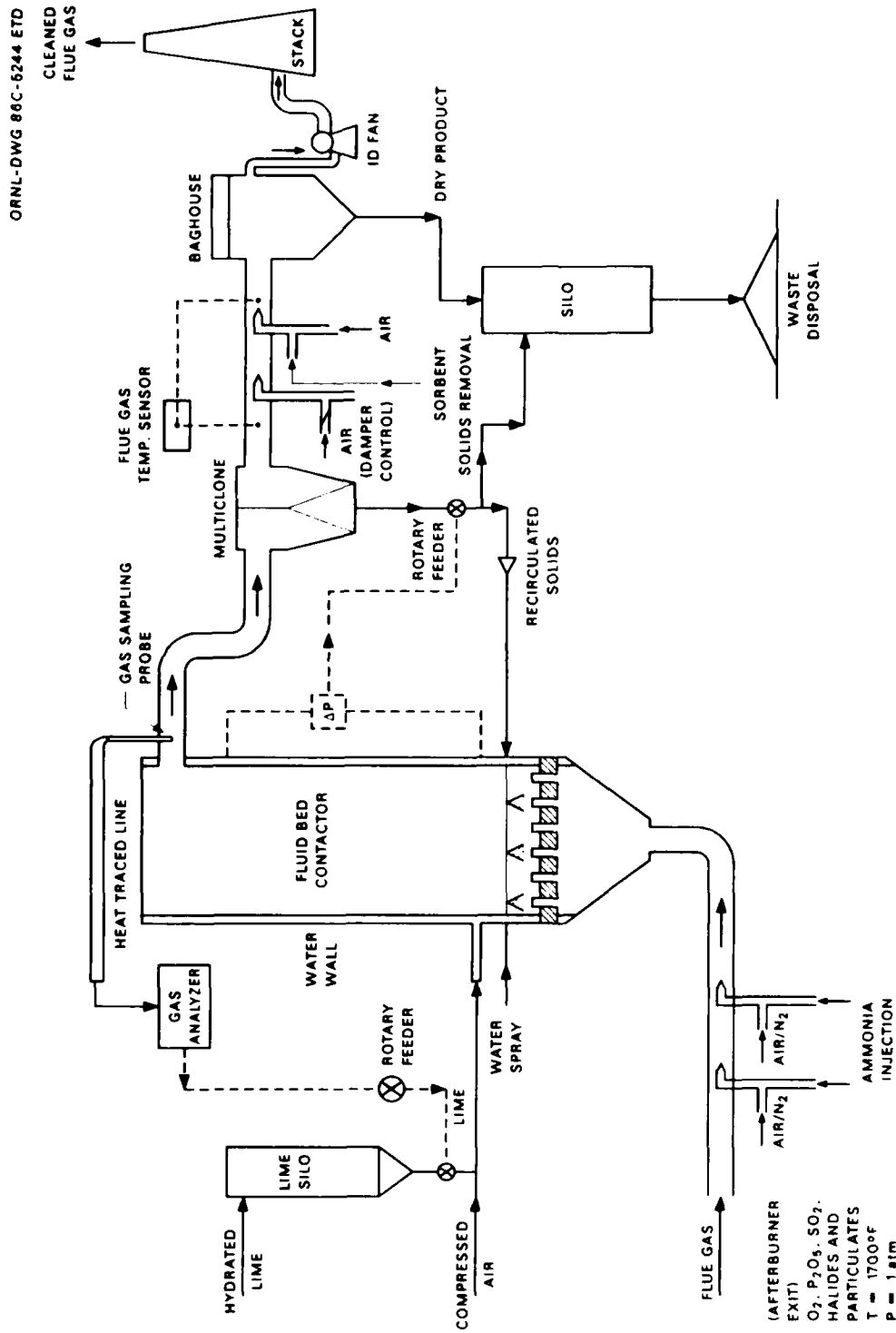


Fig. 2.1. Typical dry scrubbing process.

3. EXPERIMENTAL PROGRAM

The experimental program described in this report is part of an ongoing effort by USATHAMA to develop and test a flue gas dry scrubbing system as an alternative to wet scrubbing for Army incinerators. The bench-scale experiments are divided into two phases: the first including data needed to determine basic feasibility and summarized in this report, and the second planned for FY 1988. It will provide data necessary to determine the economic feasibility of dry scrubbing. In addition, it will provide data that will be used in designing a pilot-scale dry scrubbing system if the economic analysis proves favorable.

3.1 SCOPE

The FY 1987 portion of the program focused on scrubbing P2O5 and SO2 in a bubbling bed of quicklime. These gases were chosen because of the lack of data for P2O5 and the comparative difficulty of scrubbing SO2 in a fluidized bed. Quicklime was chosen for reasons cited in Sect. 3.3. A bubbling bed contacting scheme was chosen for its ease of operation at the bench scale.

3.2 TEST CONDITIONS

The FY 1987 experimental program consisted of 13 tests. The first eight involved scrubbing P2O5 at a variety of temperatures and flow rates. Three tests were run to verify that conditions favorable for P2O5 removal are also acceptable for SO2 scrubbing. Finally, two tests were run with P2O5 and SO2 simultaneously to determine if the presence of one affects the removal rate of the other.

The test conditions studied for P2O5 scrubbing were chosen to coincide with conditions reported in the literature that are favorable for SO2 removal.⁵ The parameters studied included bed temperature, inlet concentration, gas velocity, and humidity. The range of interest is shown in Table 3.1.

Table 3.1. Test conditions

Parameter	Range tested
Bed temperature, °C (°F)	480–650 (900–1200) for P ₂ O ₅ 540 (1000) for SO ₂ and SO ₂ /P ₂ O ₅
Inlet concentration, ppm	23–1900 for P ₂ O ₅ 2900–6600 for SO ₂
Gas velocity, cm/s	4.8–9.5 for P ₂ O ₅ 5.7–6.8 for SO ₂ 8.5 for SO ₂ /P ₂ O ₅
Humidity, % (molar basis)	54 for P ₂ O ₅ 0 for SO ₂ 0 for SO ₂ and P ₂ O ₅
Sorbent particle size, μm	212–425 for all tests

3.3 SELECTION OF SORBENT

Sorbents used in dry scrubbing processes include sodium-based sorbents, such as sodium carbonate, sodium bicarbonate, nacholite, and trona; calcium-based sorbents, such as limestone (CaCO₃), hydrated lime [Ca(OH)₂], quicklime (CaO), and dolomitic lime [Ca(OH)₂ Mg(OH)₂]; and, to a lesser degree, magnesium-based sorbents, such as magnesium oxide. Magnesium-based sorbents were not considered because there is very little data available in the literature and they are not as widely available commercially as other sorbents. Calcium-based sorbents were chosen over sodium-based sorbents because

1. lime is typically 10 to 15% of the cost of alkali materials (per molar equivalent);
2. with the exception of CaCl₂, the resulting calcium salts are not soluble in water; and
3. sodium-based sorbents are generally limited to temperatures below 370°C (700°F) because of sorbent sintering and eventual melting of the sodium compounds.

Several calcium-based sorbents were considered, including limestone, hydrated lime, quicklime, and dolomitic lime.

Limestone must first be calcined before it will readily react with most acidic gases.



The temperature required for the calcination reaction is in the range of 760° to 925°C (1400 to 1700°F), depending upon process conditions. Because the temperature range of interest was 480 to 650°C (900 to 1200°F), limestone was eliminated from further consideration.

Hydrated lime was also considered. However, at ~480°C (900°F), calcium hydroxide decomposes to form calcium oxide and water. The physical and chemical properties of hydrated lime change radically from 480 to 650°C. However, some studies have shown that hydrated lime and hydrated dolomitic lime result in higher SO₂ removal and sorbent utilization than quicklime in this temperature range.

The objective of this portion of the study was to determine the feasibility of the dry scrubbing process for removing SO₂ and P₂O₅. Physical and chemical changes in the sorbent could serve to bias the data, thus resulting in little or no benefit. Hydrated lime should not be eliminated for further study but was not chosen in this case to limit the number of variables in the process.

Quicklime was chosen because it is stable in the temperature range of interest. In addition, its fluidization characteristics proved to be the best of the sorbents tested (hydrated lime, quicklime, and dolomitic lime). The sorbent, supplied by the Tenn-Luttrell Company in Luttrell, Tennessee, was nominally 92.2% CaO, 7.4% Ca(OH)₂, and 0.5% MgO.

4. EXPERIMENTAL EQUIPMENT AND PROCEDURES

All of the bench-scale tests were conducted in the scrubber system shown in Fig. 4.1. The bubbling bed was contained in a 2-in. Schedule 40 pipe with an expansion section to reduce entrainment of solids. The bed depth was ~20 cm (8 in.).

4.1 SYSTEM DESCRIPTION

A schematic diagram of the dry scrubber system is shown in Fig. 4.2. Solid P₂O₅ was sublimed into a nitrogen stream, and the gaseous mixture flowed through a tuyere plate and entered the scrubber. Sulfur dioxide was introduced as a gas via a port immediately below the tuyere plate.

The scrubber consisted of a bubbling bed of quicklime in a 2-in. Schedule 40 pipe. The residence time in the scrubber ranged from 2.2 to 4.2 s. The effluent gas stream from the scrubber was bubbled through a series of gas-washing bottles containing water or sodium hydroxide (depending on the acidic gas being trapped). The gas-washing bottles were analyzed to determine the amount of acidic gas leaving the scrubber.

4.1.1 Fluidized Bed Scrubber

The fluidized bed scrubber section was a 2-in. Schedule 40 316 stainless steel pipe 46 cm (18 in.) long. A 4-in. Schedule 40 pipe 76 cm (30 in.) long was provided immediately above the 2-in. section to reduce entrainment of solids. Both ends of the scrubber were flanged to permit disassembly.

The acidic gas/nitrogen mixture was fed into the bottom of the scrubber and flowed through a porous metal (316 stainless steel) tuyere plate designed to enhance gas distribution and to support the lime. A small sparging tube near the bottom of the bed was used to introduce water vapor into the scrubber during some of the tests.



Fig. 4.1. Bench-scale dry scrubbing system.

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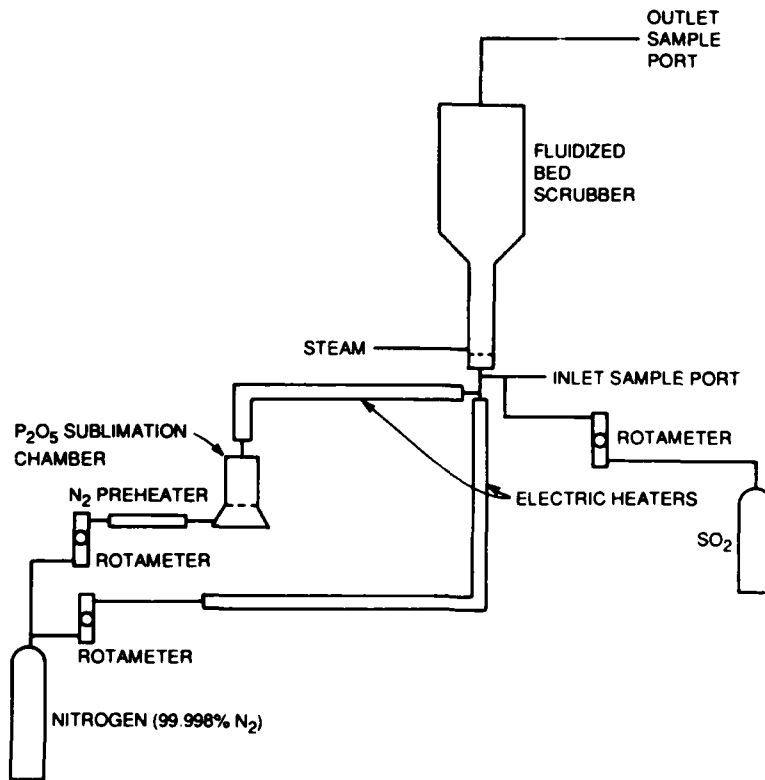


Fig. 4.2. Schematic diagram of dry scrubber.

4.1.2 Sublimation Chamber

P2O5 was loaded into a 2-in. Schedule 40 316 stainless steel pipe 15 cm long (Fig. 4.3). The inlet and outlet were fitted with porous 316 stainless steel plates to contain the P2O5 crystals. The sublimation chamber was kept as dry as possible to reduce the amount of poly-phosphoric acid formed.

Dry nitrogen (99.998% N₂) was fed into the chamber and exited nearly saturated with P2O5. The amount of P2O5 introduced into the scrubber was a function of the sublimation chamber temperature (which determines the P2O5 partial pressure) and the nitrogen flow rate through the sublimation chamber.

The stainless steel tubes and valves used to transport and control the flow of the P2O5/nitrogen mixture were maintained at a minimum of 750°F to prevent the P2O5 from condensing.

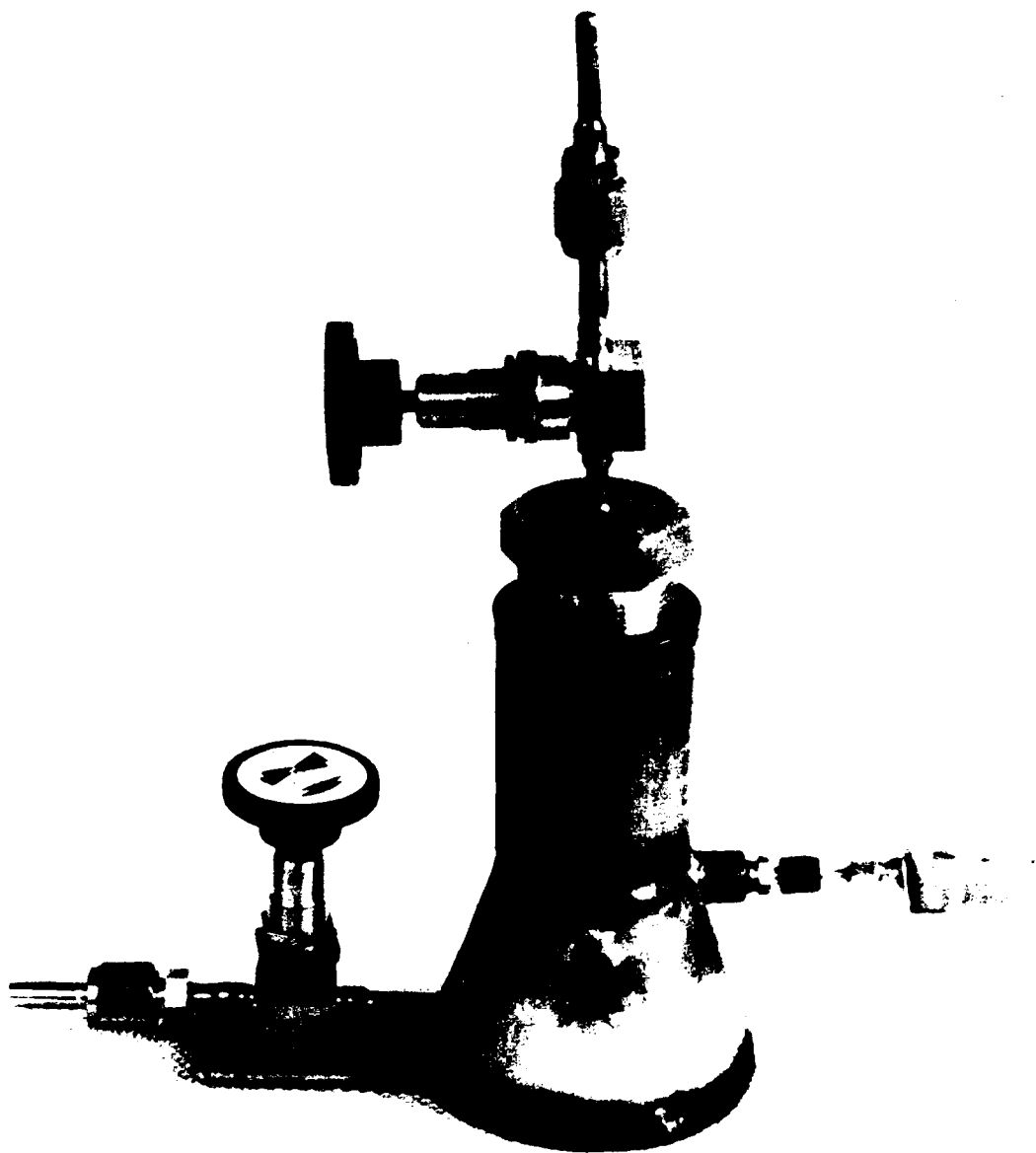


Fig. 4.3. P₂O₅ sublimation chamber.

4.1.3 Instrumentation

The bed temperature was recorded at four locations during each run. Temperatures were also monitored at selected locations to ensure that P₂O₅ did not condense in the process lines. The temperature of the

P2O5 sublimation column was controlled to produce a constant P2O5 concentration. The gas flow rates were monitored and controlled with rotameters.

4.2 SAMPLING AND DATA COLLECTION

Acidic gases exiting the scrubber were trapped in gas-washing bottles. The bottles were changed every hour to provide a series of time-weighted averages of effluent concentration of the particular gas being scrubbed. The inlet stream was sampled periodically in a similar manner to determine the inlet concentration of the acidic gas being scrubbed.

The sample train was tailored to the specific gas being sampled. Phosphorous pentoxide is extremely soluble in water, so a single gas-washing bottle filled with distilled water was used to trap the P2O5 in the outlet gas stream. Sulfur dioxide is only slightly soluble in water, but is easily scrubbed by 0.1- to 1 *N* sodium hydroxide solutions. The SO2 samples were captured in a two-stage train. The first bottle contained deionized water to cool the gas, and the second bottle contained 1.0 *N* NaOH to capture the SO2.

The sample bottles were analyzed for total phosphorous by inductive coupled plasma (ICP) emission. The total sulfur was determined by ion chromatography. At the conclusion of each test, a sample of the bed material was digested in nitric acid and analyzed for total phosphorous or total sulfur.

4.3 OPERATING PROCEDURES

Except for run 8, which was a long-term test (80 h) with P2O5, all of the tests were 1-d tests. The tests consisted of warming up the scrubber and associated equipment, bringing all of the mass flows to steady-state, and operating the scrubber. The test protocol called for sampling the gas being introduced to the scrubber before and after the run, as well as every hour during the run. Most of the tests consisted

of four 1-h samples taken at the scrubber outlet sandwiched between five 30-min samples taken at the scrubber inlet.

4.3.1 System Warm-Up

The first step in conducting a test was to bring the scrubber to operating temperature. While the scrubber was heating up, the sample lines were heated to a minimum of 400°C. The heating elements for the scrubber and P₂O₅ sublimation chamber were controlled by proportional/integral controllers. The heaters used to preheat the nitrogen were controlled by proportional controllers.

4.3.2 P₂O₅ Introduction

Once the sample lines reached 400°C, the valves at the inlet and outlet of the P₂O₅ sublimation chamber were opened, causing the P₂O₅ to be sublimed into dry nitrogen. Once the column reached operating temperature, a series of samples were taken and analyzed to ensure that the flow of P₂O₅ through the sublimator had reached steady-state. Once steady-state was reached, the first inlet sample was taken, and testing proceeded.

4.3.3 SO₂ Introduction

SO₂ flowed through a rotameter and entered the scrubber near the inlet sample port. The flow rate of SO₂ was controlled by a needle valve on the rotameter.

4.3.4 Operation

Tests typically ran 6 to 7 h. The outlet sampling bottles were changed every hour, and inlet samples were taken before and after each outlet sample.

4.3.5 Shutdown

The system was allowed to cool for 1 h under a dry nitrogen purge. After 1 h the system was pressurized to ~300 kPa (30 psig) with dry nitrogen and allowed to cool until the next day.

4.4 SAMPLE ANALYSIS

The total phosphorous in the liquid samples was determined by ICP emission, and total sulfur was determined using ion chromatography. At the conclusion of each test, a sample of lime was digested in an acidic solution and tested for total phosphorous and total sulfur, using the same ICP technique. A material balance provided a check on removal efficiency of the acidic gas being tested.

5. DISCUSSION AND RESULTS

The test program focused on determining which parameters affect the removal rate of acidic gases and the concentration of acidic gas at the scrubber outlet. The parameters studied included bed temperature, inlet concentration, humidity, and gas flow rate. The majority of tests were aimed at determining how these parameters affect the scrubbing of P_2O_5 because literature data on dry scrubbing of P_2O_5 could not be located. However, there is a significant volume of data in the literature on flue gas desulfurization and, to a lesser degree, removal by dry scrubbing of halides from a gas.

The range of test conditions for scrubbing P_2O_5 had to be determined with very little data. As a starting point, temperatures $<400^\circ\text{C}$ (750°F) were eliminated because P_2O_5 will react with water vapor to form an acid mist below 400°C . This mist is very corrosive and, moreover, probably would impede contacting between the lime particles and the gas, resulting in lower removal efficiency.

Ultimately, a dry scrubber would be expected to remove a variety of acidic gases. Economically, it is probably advantageous to remove all of the gases in the same contactor. An operating envelope of 480 to 650°C (900 to 1200°F) was chosen to coincide with conditions favorable to the removal of SO_2 in a fluidized bed. Likewise, the envelope for gas velocity of 1.2 to 3.5 times the minimum fluidization velocity was chosen because it proved favorable in other tests involving desulfurization.⁵

5.1 SUMMARY OF RESULTS

The bench-scale scrubber removed $>99\%$ of the P_2O_5 and 99% of the SO_2 at all of the conditions tested.

Eight tests were conducted to examine the effect of temperature, gas flow rate, humidity, and inlet concentration of the removal of P_2O_5 in a fluidized bed of quicklime. Of these operating parameters, the bed temperature and gas flow rate were found to have the most significant effect on scrubber performance. The P_2O_5 removal rate was higher at

540°C (1000°F), and the removal rate was also higher at 3.5 times the minimum fluidization velocity than at 1.8 times the minimum fluidization velocity. Inlet P₂O₅ concentration and humidity did not have a significant effect on the amount of P₂O₅ in the effluent stream.

Three tests were run to verify that SO₂ removal rates reported in the literature could be obtained in the bench-scale system used in this study. The removal rate and outlet concentration obtained in tests 9 and 10 compared quite well with other studies.⁶ The inlet and outlet samples taken in test 11 were saturated with SO₂, so no meaningful conclusions can be drawn from that test.

An additional two tests scrubbing P₂O₅ and SO₂ simultaneously from the carrier gas were run. Results from these tests showed that >99.7% of the P₂O₅ and >99.8% of the SO₂ could be removed from a gas containing both acidic gases.

5.2 SCRUBBER PERFORMANCE FOR P₂O₅ REMOVAL

The results of the P₂O₅/lime tests are presented in Table 5.1. The P₂O₅ removal rate was >99% at all of the conditions tested. The parameters that seem to have the greatest effect on scrubber performance are gas flow rate and temperature. The humidity did not seem to change the removal rate or outlet concentration.

The inlet P₂O₅ concentration ranged from 23 ppm in test 3 to 1900 ppm in test 7. The average P₂O₅ outlet concentration was low (0.03 to 0.7 ppm) for all of the conditions studied. The outlet concentration was more a function of gas flow rate and bed temperature than inlet concentration. For this reason, outlet concentration is more indicative of scrubber performance than removal efficiency.

The fact that outlet concentration is not strongly dependent on inlet concentration is encouraging because the concentration of acidic components in flue gas produced by an incinerator will vary widely from one feed material to another. However, operating parameters, such as bed temperature and gas flow, are relatively easy to control.

Table 5.1. Results of dry scrubbing tests

Test No.	Acid	Temperature (°C)	Gas flow rate (cm/s)	U/U_{mf}^a	Concentration (ppm)		Remarks
					Inlet	Outlet	
1	P2O5	540	4.78	1.8	92	<0.13	
2	P2O5	540	4.78	1.8	80	<0.16	
3	P2O5	650	5.43	1.8	23	<0.14	
4	P2O5	480	4.80	1.9	120	0.40	
5	P2O5	540	5.08	1.9	230	0.55	120 g steam/h
6	P2O5	540	4.87	1.9	980	0.70	
7	P2O5	540	9.18	3.5	1900	0.32	
8	P2O5	540	9.5	3.6	480	0.03	
9	SO2	540	5.69	2.2	6600	2.0	
10	SO2	540	6.79	2.6	4600	1.6	
12	P2O5 SO2	540	8.45	3.2	1500 2900	<1 6.8	
13	P2O5 SO2	540	8.45	3.2	220 3100	<1 <3.6	

^aGas velocity/minimum fluidization velocity.

The higher gas velocity (9.2 cm/s) used in test 7 resulted in the best overall performance for the short-term tests. Again, this has positive implications for the plant-scale operation because higher gas velocity results in smaller process equipment and lower capital costs.

The process tested involved the reaction between P2O5 in the gas phase and calcium oxide in the solid phase. For the reaction to take place, the P2O5 must diffuse through a boundary layer around the solid particles. The mass transfer from the gas phase to the solid phase is a function of the Sherwood number, which is related to the Schmidt and Reynolds numbers.⁷ The relationship for a single spherical particle

moving through a fluid is

$$Sh = 2.0 + 0.6 Sc^{1/3} Re_p^{1/2},$$

$$Sh = \text{Sherwood number} = \frac{k_d d_p y}{D},$$

$$Sc = \text{Schmidt number} = \frac{\mu}{\rho D},$$

$$Re_p = \text{Reynolds number} = \frac{d_p u_o \rho}{\mu}.$$

where

k_d = P2O5 mass transfer coefficient,

d_p = lime particle diameter,

y = logarithmic mean fraction of nitrogen,

D = diffusion coefficient of P2O5 in N2,

μ = viscosity of bulk gas,

ρ = density of bulk gas, and

u_o = velocity of bulk gas relative to lime particle.

The diffusion rate will increase at higher gas velocities as a result of the increase in the Reynolds number. This was observed in these tests and has been reported in tests involving the removal of SO2 from flue gas in a circulating fluidized bed.⁸

The removal rate was consistently higher at 540°C (1000°F) than at 480°C (900°F). For example, tests 2 and 4 were run at identical conditions except that the bed temperature was 540 and 480°C, respectively. The average inlet concentrations were 80 and 120 ppm, respectively. The outlet concentration at 540°C averaged <0.16 ppm (the detectable limit at those conditions); the outlet concentration at 480°C averaged 0.4 ppm. Although the difference between 0.16 and 0.4 ppm is probably not significant in terms of meeting environmental standards, it is indicative of improved removal efficiencies at higher temperatures. The inlet P2O5 concentration used in the one run at 650°C (1200°F) was too low to yield a meaningful removal rate (the outlet concentration was always below detectable limits).

5.3 SCRUBBER PERFORMANCE FOR SO₂ REMOVAL

Two successful tests, conducted at 540°C (1000°F), verified that SO₂ can be scrubbed at conditions favorable to P₂O₅ removal. The gas velocity ranged from 5.7 to 6.8 cm/s (0.19 to 0.22 ft/s); the inlet SO₂ concentration ranged from 4600 to 6600 ppm. The results of these tests are presented in Table 5.1.

In both cases, >99% of the SO₂ was removed, a finding which coincides with results in the literature.⁶ A circulating fluidized bed scrubber operating at the Schwardorf Power Station in West Germany removes 94 to 97.5% of the SO₂ at 60% lime utilization.⁹ Note that the tests run in this phase of the experimental program were conducted at <10% lime utilization. Somewhat lower removal efficiencies would be expected at higher lime utilization.

5.4 SCRUBBER PERFORMANCE FOR P₂O₅ AND SO₂ REMOVAL

Two tests were run to determine if any coeffects exist between P₂O₅ and SO₂ when they are scrubbed simultaneously. The results are presented in Table 5.1.

A significant amount of fines, of approximately the same chemical composition as the rest of the bed, was generated when the two gases were tested simultaneously. Although sorbent losses in the bed were not significant (1 to 2%), the fines plugged the outlet sampling train. In a production-scale dry scrubber the fines would be trapped by fabric filter bags, so they do not represent a significant problem once the process is scaled up. However, changes will be made to prevent plugging of the outlet line before further bench-scale tests are run.

6. CONCLUSIONS AND RECOMMENDATIONS

The results obtained from bench-scale experiments indicate that it is feasible to scrub essentially all of the P_2O_5 from a carrier gas in a bubbling bed. The experimental results, along with a large number of studies conducted by others, indicate that SO_2 also can be adequately scrubbed at conditions favorable for P_2O_5 removal. Finally, no adverse effects between P_2O_5 and SO_2 were observed when they were tested simultaneously.

Dry scrubbing is a viable technology for removing acidic gases from flue gases produced by the incineration of hazardous materials. It also appears to offer some advantages over wet scrubbing and may result in lower reagent costs and reduced capital and operating costs of treating waste products.³

Future bench-scale efforts should be aimed at

1. determining the feasibility of removing 99% of the HCl from flue gas, as required by RCRA;
2. determining scrubber performance with a mixture of acidic gases that represent a typical flue gas mixture composed of nitrogen, oxygen, carbon dioxide, hydrogen chloride, phosphorus pentoxide, sulfur dioxide, and water vapor;
3. screening candidate sorbents; and
4. obtaining data, such as ultimate sorbent utilization (relationship between removal efficiency and fraction of sorbent converted to a calcium salt), needed for an economic comparison of wet and dry scrubbing.

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Appendix A

CHARACTERIZATION OF BED MATERIAL

The sorbent used in all of the tests was commercial-grade quicklime supplied by Tenn-Luttrell. The material was sized into -212, 212 to -425, 425 to -600, and 600 μm . Screening the material resulted in approximately the same amount in each fraction. The 600- μm material was run through a hammer mill to produce more of the smaller fractions. The 212- to -425- μm fraction was used in this study because it was less prone to channeling than the finer material, had a larger specific surface area than the larger-size ranges, and required a lower gas flow rate for minimum fluidization than the larger-sized particles. The particle size distribution of the as-received lime is given in Table A.1.

Table A.1. Particle size distribution of as-received lime

Screen size (μm)	Fraction (%)
600	23
-600 to 425	24
-425 to 212	19
-212	34

The lime used in this study, the -425- to 212 μm size fraction, consisted of 54.9% below 300 μm , and 45.1% above 300 μm , yielding a harmonic mean particle size of 295 μm . The compacted bulk density of the particles was 0.93 g/cm³.

The bed material was dry. There was no detectable weight loss after drying in an oven at 120°C for 16 h. The nominal chemical analysis of the bed material (Table A.2) represents the average of several analyses run on bed material.

Table A.2. Chemical analysis
of bed material

Compound	Weight fraction (%)
Calcium oxide	92.2
Calcium hydroxide	7.4
Magnesium oxide	0.5
Total	100.1

Appendix B

BED FLUIDIZATION TESTS

A series of tests was conducted to evaluate the fluidization characteristics of potential sorbent materials. A second series of tests was conducted with a variety of lime and limestone materials to evaluate different gas distributors. Operationally, materials or gas distributors that reduce or eliminate channeling and slugging result in the most efficient gas-solid contacting and are, therefore, the most efficient in scrubbing acidic gases.

B.1 CHANNELING

Channeling is the formation of a low-density vertical path through the bed. This path has a much lower pressure drop than the remainder of the bed, so a greater fraction of the gas flows through it. The increased gas velocity increases local entrainment of solids, further reducing the pressure drop and increasing the fraction of the gas that flows through this vertical path.

The gas-solid contacting in the low-density vertical path is very poor in comparison with the rest of the bed. In addition, the residence time for the gas in the low-density path is significantly reduced. Channeling is evident because the pressure drop across the bed is typically <50% of the pressure drop without channeling.

Channeling can be reduced by proper selection and preparation of solids and by proper distributor plate design. The pressure drop across the distributor plate is typically 20 to 30% of the pressure drop across the bed. Levenspiel recommends that the pressure drop across the distributor be the maximum of (1) 10% of the bed pressure drop, (2) 100 times the pressure loss of expansion into the fluidized bed, or (3) 14 inches of water.¹

Materials that tend to agglomerate are prone to channeling. Tests conducted on 100- to -212- μ m limestone channeled. It was obvious from squeezing a handful of the limestone that the lower-size fractions of limestone would easily agglomerate. Lime particles in the 100- to

-212- μ m size range channeled slightly, but not nearly as much as limestone.

B.2 SLUGGING

Slugging is the formation at the bottom of the bed of very large bubbles that move through the bed without breaking up. The efficiency of gas-solid contacting is inversely related to the bubble size. Slugging causes the solids to spout and will cause a sinusoidal change in pressure drop across the bed as bubbles form and then move through the bed.

Slugging can be reduced by lowering the aspect ratio (the ratio of length to diameter) or changing the distributor plate. The lime particles used in this study slugged at a bed height of 30 cm (an aspect ratio of 5.7) but did not slug when the bed height was reduced to 20 cm (an aspect ratio of 3.8). There did not seem to be a relationship between particle size and slugging for the lime or limestone that was tested.

B.3 MINIMUM FLUIDIZATION VELOCITY

Fluidization behavior is determined by plotting pressure drop across the bed vs gas velocity on a log-log scale. A typical plot of ideal fluidization is shown in Fig. B.1. Slugging results in the curve presented in Fig. B.2, and channeling typically results in the relationship shown in Fig. B.3.

The minimum fluidization velocity is defined as the minimum gas velocity required to suspend the solid particles. The pressure drop across the bed is simply the sum of the pressure required to support the solid particles and the friction losses associated with gas flow. The pressure drop required to support the particles is simply the total weight of solids divided by the cross-sectional area of the bed.

Once minimum fluidization has been reached, the pressure drop across the bed increases only slightly with velocity (as a result of the

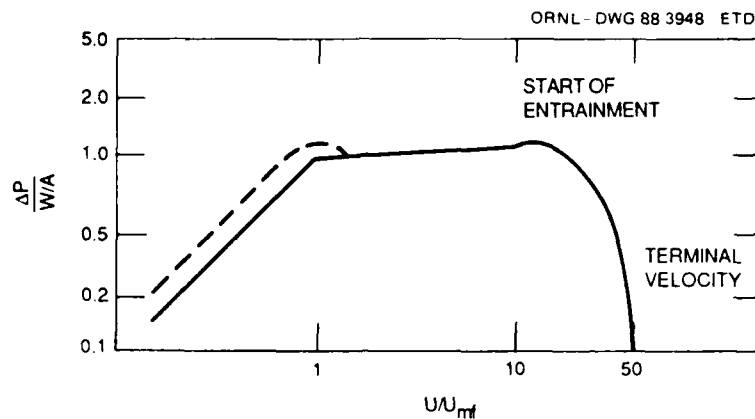


Fig. B.1. Ideal fluidization. Adapted from D. Kunni and O. Levenspiel, *Fluidization Engineering*, Robert E. Krieger Publishing Co., Inc., Huntington, NY, 1977.

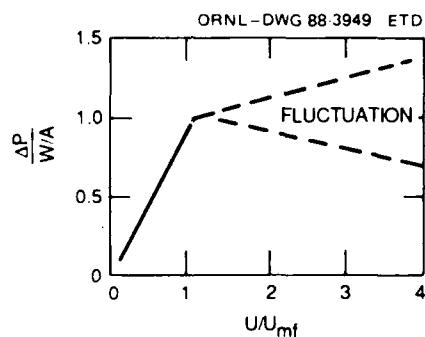


Fig. B.2. Slugging. Adapted from D. Kunni and O. Levenspiel, *Fluidization Engineering*, Robert E. Krieger Publishing Co., Inc., Huntington, NY, 1977.

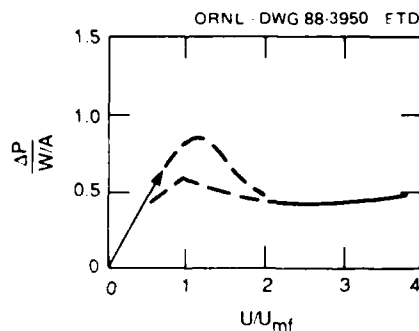


Fig. B.3. Channelling. Adapted from D. Kunni and O. Levenspiel, *Fluidization Engineering*, Robert E. Krieger Publishing Co., Inc., Huntington, NY, 1977.

increase in friction forces acting on the gas). At higher gas velocities, solids are entrained, and the pressure drop decreases because there are less solids remaining in the bed.

The minimum fluidization velocity served as the basis of determining the test conditions. Other laboratory studies on dry flue gas desulfurization have reported high acidic gas removal rates in the range of 1.8 to 3.5 times minimum fluidization velocity. This velocity meets the criteria for a vigorously bubbling bed.¹

B.4 FLUIDIZATION TESTS

The properties having the greatest effect on minimum fluidization velocity are solid and gas density, gas viscosity, harmonic mean particle size, bed voidage at minimum fluidization, and particle sphericity. The minimum fluidization velocity must be determined experimentally with the actual bed material and gas and at each temperature of interest. The minimum fluidization velocity was determined for the temperatures used in scrubbing tests (results presented in Figs. B.4-B.7).

A number of tests were run with limestone to optimize the distributor plate design. In addition, tests were run with both limestone and lime to determine what size of fractions are prone to slugging or channeling and what maximum aspect ratio can be used.

Both the lime and limestone were prone to channeling with a distributor plate that used 21 high-velocity orifices to obtain 2 to 3 in. of water pressure drop. Porous metal plates provided the necessary pressure drop but produced an even flow of small bubbles rather than a limited number of high-velocity streams. The pore sizes that worked best for this application were 2 and 5 μm .

Slugging was not a problem at aspect ratios of four and below in the 2-in. bed using 212- to 425- μm particles. Both the lime and limestone tended to slug at aspect ratios above five.

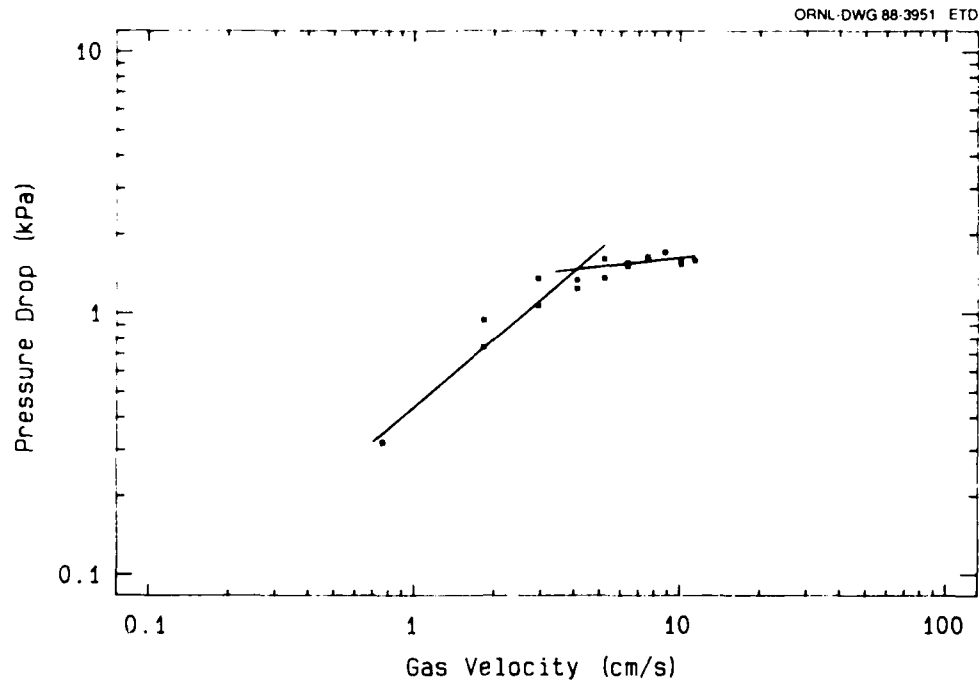


Fig. B.4. Pressure drop vs gas velocity for 212- to 425- μ m lime at 24°C, $U_{mf} = 4.1$ cm/s.

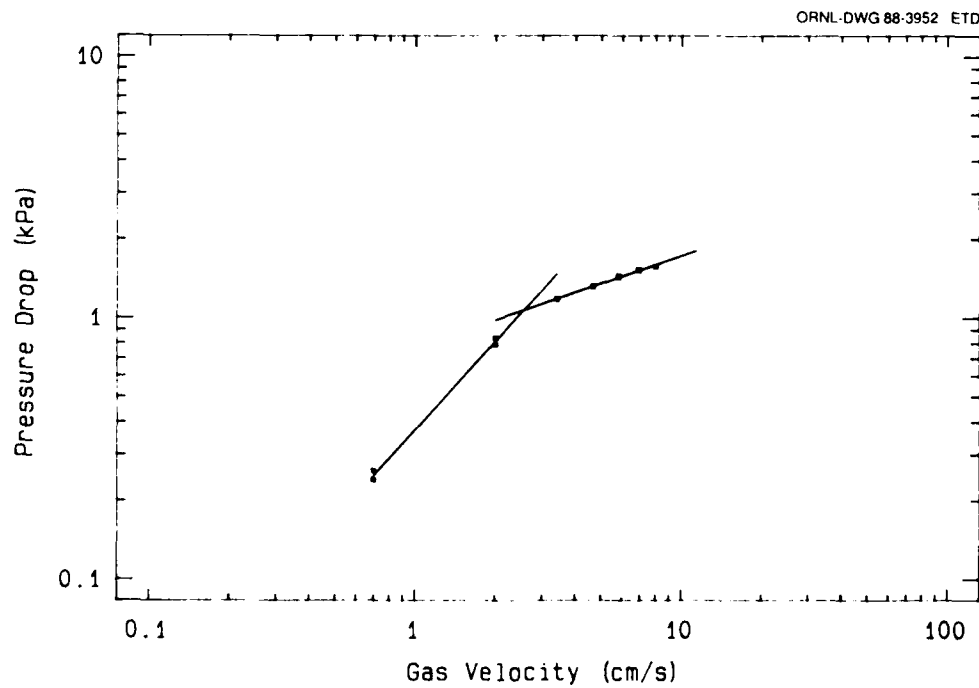


Fig. B.5. Pressure drop vs gas velocity for 212- to 425- μ m lime at 485°C, $U_{mf} = 2.6$ cm/s.

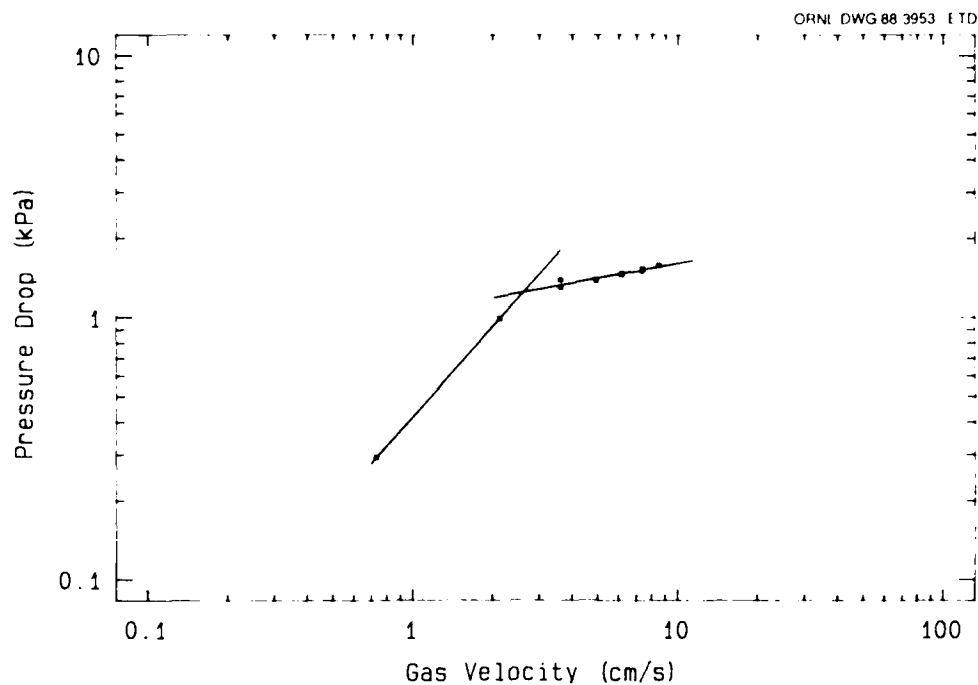


Fig. B.6. Pressure drop vs gas velocity for 212- to 425- μ m lime at 534°C, $U_{mf} = 2.6$ cm/s.

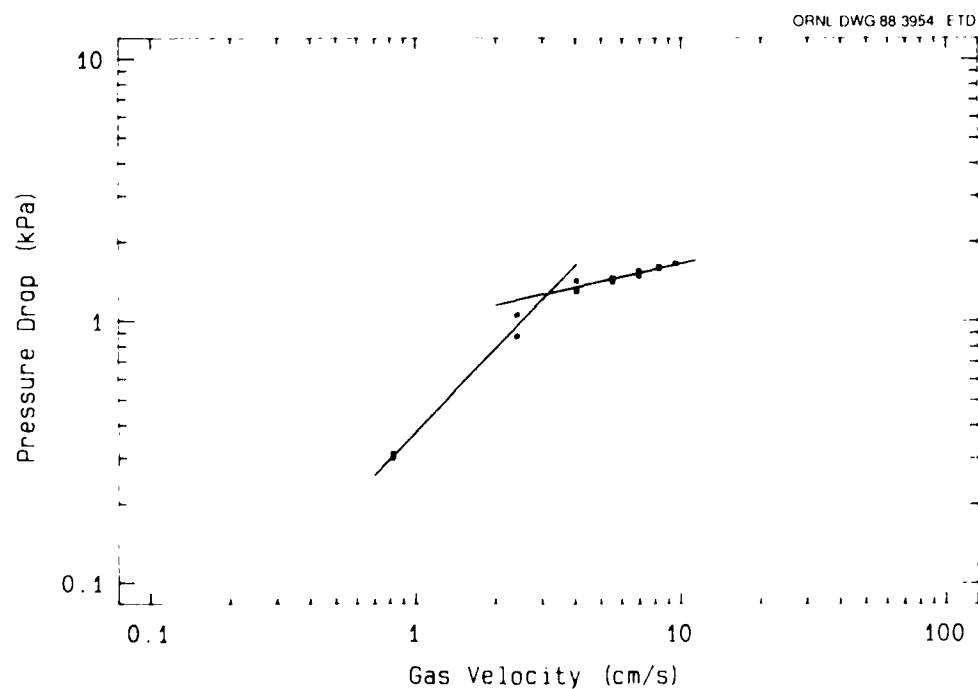


Fig. B.7. Pressure drop vs gas velocity for 212- to 425- μ m lime at 642°C, $U_{mf} = 3.2$ cm/s.

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Appendix C

EXPERIMENTAL DATA

The data obtained in each of the tests are presented in Table C.1. The test protocol called for alternating inlet and outlet samples. The typical test run started with a 30-min sample at the inlet of the scrubber, followed by 60 min of feeding the acidic gas to the scrubber, followed by another inlet sample, etc. In all, the typical test involved five inlet samples and four 1-h samples at the scrubber outlet.

Table C.1. Experimental data

Test No. 1

Acid: P₂O₅

Bed Temperature: 540°C

N₂ Flow: 5.46 mol/h

Gas Velocity: 4.78 cm/s

Total Lime: 400 g

Sample No.	Location	Time (min)	P ₂ O ₅ flow (10 ⁻⁶ mol/h)	P ₂ O ₅ concentration (ppm)
1-1	Inlet	60	178	33
1-2	Outlet	60	<0.73	<0.14
1-3	Outlet	60	<0.73	<0.14
1-4	Outlet	60	<0.71	<0.13
1-5	Outlet	60	<0.71	<0.13
1-6	Inlet	60	823	151
1-8	Lime before test 670 µg P/g			
1-7	Lime after test 1,290 µg P/g			

Table C.1. (continued)

Test No. 2

Acid: P₂O₅

Bed Temperature: 540°C

N₂ Flow: 5.46 mol/h

Gas Velocity: 4.78 cm/s

Total Lime: 400 g

Sample No.	Location	Time (min)	P ₂ O ₅ flow (10 ⁻⁶ mol/h)	P ₂ O ₅ concentration (ppm)
2-1	Inlet	30	197	36
2-2	Outlet	60	<0.76	<0.14
2-3	Inlet	30	622	114
2-4	Outlet	60	<0.71	<0.13
2-5	Inlet	30	618	113
2-6	Outlet	60	<0.76	<0.14
2-7	Inlet	30	524	9.6
2-8	Outlet	60	1.2	0.22
2-9	Inlet	30	215	39
1-8	Lime before test			670 µg P/g
2-10	Lime after test			1,100 µg P/g

Test No. 3

Acid: P₂O₅

Bed Temperature: 650°C

N₂ Flow: 5.46 mol/h

Gas Velocity: 5.43 cm/s

Total Lime: 400 g

Sample No.	Location	Time (min)	P ₂ O ₅ flow (10 ⁻⁶ mol/h)	P ₂ O ₅ concentration (ppm)
3-1	Inlet	30	65.3	12
3-2	Outlet	60	<0.71	<0.13
3-3	Inlet	30	78.1	18
3-4	Outlet	60	<0.76	<0.14
3-5	Inlet	30	159	29
3-6	Outlet	60	<0.77	<0.14
3-7	Inlet	30	149	27
3-8	Outlet	60	<0.87	<0.16
3-9	Inlet	30	149	27
1-8	Lime before test			670 µg P/g
3-10	Lime after test			980 µg P/g

Table C.1. (continued)

Test No. 4

Acid: P₂O₅
 Bed Temperature: 480°C
 N₂ Flow: 5.89 mol/h
 Gas Velocity: 4.80 cm/s
 Total Lime: 400 g

Sample No.	Location	Time (min)	P ₂ O ₅ flow (10 ⁻⁶ mol/h)	P ₂ O ₅ concentration (ppm)
4-1	Inlet	30	240	41
4-2	Outlet	60	1.7	0.29
4-3	Inlet	30	320	54
4-4	Outlet	60	5.6	0.95
4-5	Inlet	30	1,500	250
4-6	Outlet	60	1.7	0.28
4-7	Inlet	30	900	150
4-8	Outlet	60	0.9	0.15
4-9	Inlet	30	570	97
4-11	Lime before test 510 µg P/g			
4-10	Lime after test 1,500 µg P/g			

Test No. 5

Acid: P₂O₅
 Bed Temperature: 540°C
 N₂ Flow: 5.81 mol/h
 Gas Velocity: 5.08 cm/s
 Total Lime: 400 g

Sample No.	Location	Time (min)	P ₂ O ₅ flow (10 ⁻⁶ mol/h)	P ₂ O ₅ concentration (ppm)
5-1	Inlet	30	290	50
5-2	Outlet	60	1.4	0.24
5-3	Inlet	30	350	60
5-4	Outlet	60	<6.8	<1.2
5-5	Inlet	30	1,660	280
5-6	Outlet	60	1.2	0.21
5-7	Inlet	30	3,200	550
4-11	Lime before test 510 µg P/g			
5-8	Lime after test 4,500 µg P/g			

Table C.1. (continued)

Test No. 6

Acid: P2O5

Bed Temperature: 540°C

N2 Flow: 5.57 mol/h

Gas Velocity: 4.87 cm/s

Total Lime: 400 g

Sample No.	Location	Time (min)	P2O5 flow (10 ⁻⁶ mol/h)	P2O5 concentration (ppm)
6-1	Inlet	30	5,700	1,000
6-2	Outlet	60	1.6	0.29
6-3	Inlet	30	5,400	1,000
6-4	Outlet	60	3.6	0.65
6-5	Inlet	30	5,800	1,000
6-6	Outlet	60	4.7	0.85
6-7	Inlet	30	5,800	1,000
6-8	Outlet	60	6.0	1.1
6-9	Inlet	30	5,100	910
4-11	Lime before test 510 µg P/g			
6-11	Lime after test 4,500 µg P/g			

Test No. 7

Acid: P2O5

Bed Temperature: 540°C

N2 Flow: 10.5 mol/h

Gas Velocity: 9.18 cm/s

Total Lime: 400 g

Sample No.	Location	Time (min)	P2O5 flow (10 ⁻⁶ mol/h)	P2O5 concentration (ppm)
7-1	Inlet	15	32,600	3,100
7-2	Outlet	60	2.0	0.19
7-3	Inlet	15	22,600	2,200
7-4	Outlet	60	2.2	0.21
7-5	Inlet	15	9,400	890
7-6	Outlet	60	9.0	0.86
7-7	Inlet	15	5,400	510
7-8	Outlet	60	1.9	0.18
7-9	Inlet	15	30,900	2,900
7-10	Outlet	60	1.5	0.14
7-11	Inlet	15	16,700	1,600
6-10	Lime before test 990 µg P/g			
7-12	Lime after test 7,450 µg P/g			

Table C.1. (continued)

Test No. 8

Acid: P2O5

Bed Temperature: 540°C

N2 Flow: 10.4 mol/h

Gas Velocity: 9.5 cm/s

Total Lime: 400 g

Sample No.	Location	Time (min)	P2O5 flow (10 ⁻⁶ mol/h)	P2O5 concentration (ppm)
8-1	Inlet	30	4,300	420
8-2	Inlet	30	7,200	690
8-3	Outlet	240	0.34	0.03
8-4	Inlet	30	22,000	2,100
8-5	Outlet	240	0.38	0.04
8-6	Inlet	30	13,000	1,300
8-8	Inlet	30	16,000	1,600
8-9	Outlet	240	<0.23	<0.02
8-10	Inlet	30	1,700	140
8-11	Outlet	240	<0.17	<0.02
8-12	Inlet	30	1,400	140
8-13	Inlet	30	5,000	480
8-14	Outlet	240	0.68	<0.06
8-15	Inlet	30	5,300	490
8-16	Outlet	240	<0.17	<0.02
8-17	Inlet	30	3,700	350
8-18	Outlet	240	0.16	0.015
8-19	Inlet	30	3,400	330
8-20	Outlet	240	0.54	0.05
8-21	Inlet	30	3,000	290
8-22	Outlet	240	0.17	0.02
8-23	Inlet	30	2,900	300
8-24	Outlet	240	0.99	0.09
8-25	Inlet	30	2,300	210
8-26	Outlet	240	0.49	0.05
8-27	Inlet	30	2,200	160
8-28	Outlet	240	<0.16	<0.02
8-29	Inlet	30	1,400	130
8-30	Outlet	240	0.42	0.04
8-31	Inlet	30	1,400	140
8-32	Outlet	240	<0.14	<0.02
8-33	Inlet	30	950	92
8-34	Inlet	30	1,700	160
8-35	Outlet	240	<0.16	<0.02
8-36	Inlet	30	1,800	180
7-13	Lime before test 1,200 µg P/g			
8-37	Lime after test 32,000 µg P/g			

Table C.1. (continued)

Test No. 9

Acid: SO₂
 Bed Temperature: 540°C
 N₂ Flow: 5.46 mol/h
 Gas Velocity: 5.69 cm/s
 Total Lime: 400 g

Sample No.	Location	Time (min)	SO ₂ flow (10 ⁻⁶ mol/h)	SO ₂ concentration (ppm)
9-1	Inlet	30	29,300	4,500
9-2	Outlet	60	10	1.6
9-3	Inlet	30	43,200	6,700
9-4	Outlet	60	10	1.6
9-5	Inlet	30	38,000	5,900
9-6	Outlet	60	10	1.6
9-7	Inlet	30	51,000	7,800
9-8	Outlet	60	23	3.6
9-9	Inlet	30	53,000	8,200
9-11	Lime before test 860 µg S/g			
9-10	Lime after test 16,000 µg S/g			

Test No. 10

Acid: SO₂
 Bed Temperature: 540°C
 N₂ Flow: 5.46 mol/h
 Gas Velocity: 6.79 cm/s
 Total Lime: 400 g

Sample No.	Location	Time (min)	SO ₂ flow (10 ⁻⁶ mol/h)	SO ₂ concentration (ppm)
10-1	Inlet	30	50,000	6,400
10-2	Outlet	60	10	1.3
10-3	Inlet	30	33,000	4,300
10-4	Outlet	60	10	1.3
10-5	Inlet	30	27,000	3,500
10-6	Outlet	60	10	1.3
10-7	Inlet	30	42,000	5,500
10-8	Outlet	60	21	2.7
10-9	Inlet	30	25,000	3,200
10-11	Lime before test 580 µg S/g			
10-10	Lime after test 18,000 µg S/g			

Table C.1. (continued)

Test No. 12

Acid: P2O5 and SO2
 Bed Temperature: 540°C
 N2 Flow: 9.66 mol/h
 Gas Velocity: 8.45 cm/s
 Total Lime: 400 g

Sample No.	Location	Time (min)	SO2 flow (10 ⁻⁶ mol/h)	SO2 concentration (ppm)	P2O5 flow (10 ⁻⁶ mol/h)	P2O5 concentration (ppm)
12-1	Inlet	30	13,000	1,400	1,600	170
12-2	Outlet	60	100	10	<9.7	<1.0
12-3	Inlet	30	36,000	3,700	440	46
12-4	Outlet	60	44	4.5	<9.7	<1.0
12-5	Inlet	30	36,000	3,800	200	21
12-6	Outlet	60	85	8.8	<6.5	<0.7
12-7	Inlet	30	23,000	2,400	230	24
12-8	Outlet	60	35	3.6	<9.0	<1.0
12-9	Inlet	30	33,000	3,400	230	24
13-11	Lime before test 6,000 µg S/g, 770 µg P/g					
12-10	Lime after test 15,000 µg S/g, 9,900 µg P/g					

Test No. 13

Acid: P2O5 and SO2
 Bed Temperature: 540°C
 N2 Flow: 9.66 mol/h
 Gas Velocity: 8.45 cm/s
 Total Lime: 400 g

Sample No.	Location	Time (min)	SO2 flow (10 ⁻⁶ mol/h)	SO2 concentration (ppm)	P2O5 flow (10 ⁻⁶ mol/h)	P2O5 concentration (ppm)
13-1	Inlet	30	29,000	3,000	210	22
13-2	Outlet	60	<37	<3.9	<11	<1
13-3	Inlet	30	33,000	3,500	210	22
13-4	Outlet	60	<28	<2.9	<8.4	<1
13-5	Inlet	30	6,900	720	260	27
13-6	Outlet	60	<37	<3.9	<11	<1
13-7	Inlet	30	20,000	2,100	230	24
13-8	Outlet	60	<34	<3.5	<10	<1
13-9	Inlet	15	60,000	6,200	230	24
13-11	Lime before test 6,000 µg S/g, 770 µg P/g					
13-12	Lime after test 20,000 µg S/g, 2,100 µg P/g					

Appendix D

ANALYTICAL EQUIPMENT AND PROCEDURES

The inlet and outlet gas samples were captured in 100-mL gas-washing bottles. Deionized water was used for the P₂O₅ samples; sodium hydroxide solutions ranging from 0.5 to 1.0 *N* (depending on conditions) were used to capture the SO₂. The sampling system was tested to ensure that none of the acid exited the sampling train.

The solids were analyzed before and after each test to determine the amount of acid that reacted with the lime. They were digested in a nitric acid solution according to Martin Marietta Energy Systems Procedure 0908.

The aqueous samples from the P₂O₅ tests were analyzed for total phosphorous with an ICAP model 9000 spectrometer from Allied Analytical Systems. The instrument was operated in accordance with Martin Marietta Energy Systems procedure 0801.

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